SOUNDNESS OF AGGREGATE BY USE OF SODIUM SULFATE OR MAGNESIUM SULFATE
FOP FOR AASHTO T 104

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Scope

Aggregate samples are subjected to alternate cycles of immersion in soundness solution (sodium or magnesium sulfate) and drying in an oven at a regulated temperature of 230 ±9° F.

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Significance

- During the 16 to 18-hour immersion phase, salts penetrate the permeable void spaces of the aggregates.
- During the drying phase, the salt solution on the surface and within the permeable pores of the aggregate particles dehydrates resulting in an expansive pressure. This pressure simulates the expansion of water when frozen.

Apparatus

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- **Balance:** Sufficient capacity for the principal sample masses to be determined, sensitive to 0.1 g
- **Sieves:** Woven wire-cloth sieves with square openings, conforming to the requirements of AASHTO M 92.

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- **Sample Containers:** Sieves 8 inch diameter for each individual size fraction of aggregate being tested (No. 8 size for coarse aggregate, No. 60 size for fine aggregate). Sieves out of tolerance with AASHTO M 92, in an acceptable condition, may also be used as sample containers. Other types of sample containers may be used provided that they permit free access of the solution to the samples and draining of the solution from the samples without loss of aggregate. (See Note 1)

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- **Apparatus for immersing samples in solution:** Optional, for permitting free access of solution to sample and to provide for free drainage of solutions.

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- **Temperature regulation:** Suitable means for providing temperature regulation of the samples (68.5 to 71.5° F) during immersion in solution.
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Note 1: AASHTO T 104 requires the use of 8-inch diameter sieves as sample containers for referee testing, comparison testing, or for testing of aggregates to be used in critical applications.

- **Drying Oven:** Capable of maintaining heat level of 230 ±9° F for drying phase. Oven must also be capable of maintaining an evaporation rate of 25 grams per hour over a four-hour period (see "Determining Evaporation Rate").

08 - **Temperature Recorder:** Capable of recording solution temperature a minimum of once every 10 minutes throughout the test with an accuracy of 0.5° F.

09 - **Hydrometers:** Conforming to ASTM E 100. Used to check the specific gravity of the solution. Hydrometers should be capable of measuring the solution specific gravity within ±0.001. In lieu of hydrometers, AASHTO T 104 allows the use of graduated glassware and a scale to determine the specific gravity of the solutions.

10 - **Prepared Soundness Solution:** Magnesium sulfate or sodium sulfate, depending upon the procedure being used.

- **Barium Chloride Solution:** 0.2 molar solution of barium chloride to test for presence of magnesium or sodium sulfate in the final wash water.

Determining Evaporation Rate

11 1. Prepare five 1-liter Griffin Low-form beakers for each shelf of the oven. Beakers will be placed in the center and at each corner of oven shelves.
2. Place 500 g water in each beaker at 70 ±3° F. Place beakers on shelves in oven regulated at 230 ±9° F for 4 hours. Do not open the oven door during the evaporation test. Oven vents must be open to allow for venting of evaporated moisture.

12 3. At the end of the four-hour period, remove beakers and record weight of water remaining in them. The water weight in each beaker must be no more than 400 grams as per the 25-gram per hour required evaporation rate. That is, each beaker must lose at least 100 grams of water after 4 hours in an oven regulated at 230 ±9° F.

Solution Preparation

13 1. Solutions need to be prepared well in advance of the test procedure. Enough solution should be prepared to be at least 5 times the solid volume of all the samples to be tested.

2. Prepared a saturated solution of either sodium sulfate (Na_2S0_4) or magnesium sulfate ($MgS0_4$) using a reagent grade of salt added to water. Distilled water is not required but shall be used in referee or comparison testing.

3. Add enough salt to the water to ensure not only saturation but also the presence of excess crystals when the solution is ready for use in the tests. This can be accomplished by continually adding salts to the water and stirring. Add salts in increments allowing it to dissolve to determine crystallization potential.

Note 2: For the sodium sulfate solution, 225 g per liter of solution is recommended to achieve the saturated state. For magnesium sulfate solution, 350 g of anhydrous salt per liter of solution is recommended to achieve the saturated state.

14 4. Cover solution containers while not in use to prevent evaporation and contamination. Allow the solution to cool to 68.5 to 71.5° F.

5. Stir the solution and allow it to stand at least 48 hours, prior to use. Break up salt cakes, if any, prior to submerging the samples in the solution.

6. Check and record specific gravity of the solution and verify the temperature recorder is functioning each day the test is run. The temperature required for both solutions is 68.5 to 71.5° F. The specific gravity of the sodium sulfate solution must be between 1.154 and 1.171; that of the magnesium sulfate solution must be between 1.297 and 1.306.

Note 3: Discolored solution shall be discarded or filtered and checked for specific gravity.

Table 4-1

Passing Sieve	Retained on Sieve
3/8”	No. 4
No. 4	No. 8
No. 8	No. 16
No. 16	No. 30
No. 30	No. 50

Sample Sizes

15 **Fine Aggregate**

16 The sample should be passed through a 3/8 inch sieve. Sample should be of sufficient size to yield not less than 100 grams for each of the sizes shown in Table 4-1.

If the sample being tested contains less than 5% of any of the specified sizes, that size shall not be tested.

Coarse Aggregate

17 1. Remove all particles passing the No. 4 sieve prior to test.

Table 4-2

Sieve Size	Mass, g
2½" to 1½" consisting of:	5000 ± 300
2" to 1½"	2000 ±200
2½" to 2"	3000 ±300
1½" to ¾" consisting of:	1500 ±50
1" to ¾"	500 ±30
1½" to 1"	1000 ±50
¾" to ⅜" consisting of:	1000 ±10
½" to ⅜"	330 ±5
¾" to ½"	670 ±10
⅜" to No. 4	300 ±5

Coarse Aggregate

17 1. Remove all particles passing the No. 4 sieve prior to test.

18 2. The sample should be of sufficient size to yield portions conforming to the mass requirements shown in Table 4-2.

Special consideration for coarse aggregate sample preparation

19 a. If the sample contains less than 5% of any of the prescribed sieve sizes, that size should not be tested. When a combination of sizes is specified and one of the sizes specified is less than 5%, reduce the test portion by the applicable mass listed in the table.

20 b. When testing large rock (broken stone, ledge rock, cobbles, and boulders for use as riprap, channel lining, etc.), obtain the test portion by crushing, splitting, or sawing. Test only those particles in the 2½ to 1½ inch and 1½ to ¾ inch size fractions when reduction is by crushing or splitting. Test 2½ to 1½ inch size fractions when reduction is by sawing.

21 c. When testing large rock that will be crushed, obtain the test portion by crushing the aggregate. Test pieces only in those sizes that will be included in the test aggregate, but ignore any material finer than the No. 4 or larger than the 2½ inch sieve.

22 d. When the finished aggregate will contain pieces larger than 2½ inch, crush those larger pieces and distribute the crushed material among the range of 2½ inch to No. 4 sieves.

23 e. When an aggregate being tested contains appreciable amounts of both fine and coarse material (more than 10% coarser than 3/8 inch, and more than 10% finer than the No. 4 sieves), test separate samples of the coarse and fine particles in accordance with the applicable procedures, giving the percentages of fine and coarse aggregates in the original grading.

Sample Preparation

Fine Aggregate

24 1. Thoroughly wash the sample of fine aggregate on a No. 50 sieve, and dry to a constant mass at 230 ±9° F.

2. Separate the dried sample into the different sizes by sieving as required. Make a rough separation of the sample using the appropriate sieves. Obtain enough material to yield at least 100 g material on each sieve.

Note 4: After sieving to refusal, when removing the material retained on the sieves, exclude those particles stuck in the sieve mesh from the sample.

25 3. Sieve the rough-cut sample to refusal. Sieving to refusal means that no more material will pass

through the sieves after additional agitation.

4. Weigh out the samples consisting of 100.0 ±0.1 g from each of the separate fractions after final sieving. Record the mass of the test samples and place in separate containers for the test.

Coarse Aggregate

1. Thoroughly wash and dry the samples of coarse aggregate to a constant mass at 230 ±9° F.
2. Separate the sample into the different sizes according to Table 4-2 by sieving to refusal. Record the mass of the test samples to the nearest 1 g.
3. In the case of particles larger than ¾-inch, record the number of particles in each test fraction.

Procedure

1. Immerse the samples in the prepared solution for 16 to 18 hours. The solution should cover the samples to a depth of at least 1/2 inch. Cover the soundness tanks to reduce evaporation and contamination. Maintain sample temperature between 68.5 and 71.5° F.
2. After the soaking period, remove samples from the solution and allow them to drain for 15 ±5 minutes.
3. After draining, place samples in an oven regulated at 230 ±9° F. Dry the samples until a constant mass has been obtained.

Determination of Constant Mass

a. With the oven containing the maximum sample load expected, check the mass loss of test samples by removing them and weighing without cooling at intervals of 2 to 4 hours.

b. Make enough measurements to determine the drying time for the least favorable oven location and sample condition. Constant mass is achieved when mass loss is less than 0.1% in four hours of drying.

4. After achieving constant mass, allow the samples to cool to 68 to 77° F. Cooling may be aided by the use of an air conditioner or fan. Check the temperature of the material with a thermometer or other acceptable means. After cooling, immerse the samples in the solution.

5. Repeat the process of immersing, drying, and cooling until the required number of cycles has been completed. The test should be performed continuously until all cycles are completed. If the test must be interrupted, leave the samples in the oven at 230 ±9° F until testing can be resumed.

Note 5: Begin qualitative examination of the particles larger than ¾ inch before washing, it may not be possible to categorize the types of distress after the particles have been disturbed.

6. Perform the qualitative examination of aggregate particles larger than ¾ inch and record the number and percent of particles showing each of the following types of distress: disintegration, splitting, crumbling, cracking, and flaking.

7. Once all cycles have been completed, and the sample is cooled, wash the salt solution from the sample by circulating 110 ±10° F water through the samples and their containers. Introduce the warm water near the bottom and allow it to pass through the samples and overflow.

Note 6: In areas where the water gives a reaction with barium chloride, other analytical means shall be used to assure thoroughness of washing.

8. Check washing thoroughness by obtaining a sample of the rinse water after it has overflowed the samples and check with 0.2 molar barium chloride. Further washing is needed if the barium chloride causes the water to become cloudy.

9. After the solution has been completely removed from the aggregate samples, dry each fraction of the sample to constant mass at 230 ±9° F.

Note 7: When performing this final sieving of the fine aggregate particles, stack the sieves in "reverse" order with the smallest sieve on top and the largest sieve on bottom.

10. Sieve the fine aggregate over the same sieve on which it was retained before the test. Use the same mechanism and duration of sieving as for the original sample preparation.

11. Sieve the coarse aggregate particles in the fractions over the appropriate sieves shown in Table 4-3. Sieve particles by hand, with agitation sufficient only to assure that all undersized material passes the designated sieve.

Table 4-3

Size of Aggregate Fraction	Sieve Used to Determine Loss
2½" to 1½"	1¼"
1½" to ¾"	5/8"
¾" to ⅜"	5/16"
⅜" to No. 4	No. 5

Table 4-3

Size of Aggregate Fraction	Sieve Used to Determine Loss
2½" to 1½"	1¼"
1½" to ¾"	5/8"
¾" to ⅜"	5/16"
⅜" to No. 4	No. 5

Note 8: The sieves must be cleaned out completely when performing "final" sieving.

11. Sieve the coarse aggregate particles in the fractions over the appropriate sieves shown in Table 4-3. Sieve particles by hand, with agitation sufficient only to assure that all undersized material passes the designated sieve. No extra manipulation should be employed to break up particles.
12. Determine and record the mass of the particles retained on each sieve. Calculate percent loss and weighted percent loss for each size fraction.
13. Review the temperature record to verify solution temperature limits were not exceeded.

Calculations

- Perform calculations according to the formulas on the following page.
- To calculate the weighted average, use the average loss of the next larger and next smaller size for any size fraction with less than 5% of the total sample, if one of these sizes is not present use the loss of the next larger or next smaller.
- Calculate values for Percent Loss and Weighted Percent Loss of individual size fractions to the nearest 0.1%, but report total weighted percent loss of the entire sample to the nearest whole percent (See sample worksheet and report on pages 4-10 and 4-11 respectively).

Calculation Formulas and Examples (See sample worksheet on page 4-10 for data)

Quantitative Examination (Examples are for the No. 8 to No. 16 Size Fraction)

Percent Loss for Size Fraction:

$$D = \frac{B - C}{B} * 100 \quad (44)$$

$$D = \frac{99.9 - 91.9}{99.9} * 100 = 8.01, \text{ say } 8.0\% \quad (45)$$

Weighted Percent Loss for Size Fraction:

$$E = D * \frac{A}{100} \quad (46)$$

$$E = 8.0 * \frac{17}{100} = 1.36, \text{ say } 1.4\% \quad (47)$$

where:

A = individual percent retained in sample grading
B = mass of test fraction before test
C = mass of test fraction after test
D = percent loss for size fraction
E = weighted percent loss for size fraction

Qualitative Examination (Example is for Cracking in the 2½ to 1½ inch Fraction)

Percent Distressed Particles (P) in Each Category for Each Fraction Larger than ¾ inch:

$$P = \frac{\text{Number of Distressed Particles}}{\text{Number of Particles in Fraction}} * 100 \quad (48)$$

$$P = \frac{2}{29} * 100 = 6.8, \text{ say } 7\% \quad (49)$$

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Report

- Report on standard agency forms (See sample report on page 4-11)
- Date and Project
- Material source and description
- Type of solution and number of cycles
- Grading of original sample
- Mass of test fractions before test
- Percent passing the designated sieve after test
- Weighted percent loss for each fraction, based on the original sample gradation
- Total weighted percent loss for the entire sample, based on original gradation
- Qualitative examination results of coarse aggregate particles larger than the ¾ inch sieve

Tips!

- Determine aggregate gradation first
- Check solution specific gravities daily
- Record number of particles in each size fractions larger than ¾ inch prior to test
- Be sure samples have achieved constant mass during drying
- Use barium chloride to check wash water during removal of salt solution
- For coarse aggregate, check sieve size used to determine loss
- To determine loss for fine aggregate, use same sieve sizes, shaker, and sieving duration as for sample preparation
- Remember that the reported weighted losses are calculated according to the original sample gradation

Sample Worksheet

Date: _____ Project: _____

Material: _____ Solution: _____ No. of Cycles: ____

Source: _____ Tested By: _____

Quantitative Examination					
Sieve Size	Grading of Sample, %	Fraction Mass, g		Percent Loss	Weighted % Loss
		Before	After		
	A	B	C	D	E
Minus No. 100	6	---	---	---	---
No. 50 to No. 100	11	---	---	---	---
No. 30 to No. 50	26	100.0	95.8	4.2	1.1
No. 16 to No. 30	25	100.0	95.2	4.8	1.2
No. 8 to No. 16	17	99.9	91.9	8.0	1.4
No. 4 to No. 8	11	100.0	88.8	11.2	1.2
3/8" to No. 4	4	---	---	11.2	0.4
Totals	100	---	---	---	5
2½" to 1½" 2½" – 2" (2825 g) 2" – 1½" (1958 g) Total:	20	4783	4554	4.8	1.0
1½" to ¾" 1½" – 1" (1012 g) 1" – ¾" (513 g) Total:	45	1525	1402	8.1	3.6
¾" to ⅜" ¾" – ½" (675 g) ½" – ⅜" (333 g) Total:	23	1008	912	9.5	2.2
⅜" to No. 4 298 g	12	298	265	11.1	1.3
Totals	100	---	---	---	8

Qualitative Examination of Coarse Particles											
Sieve Size	Disinteg		Splitting		Crumbling		Cracking		Flaking		No. of Particles in Fraction
	No.	%	No.	%	No.	%	No.	%	No.	%	
2½" - 1½"	---	---	2	7	---	---	2	7	---	---	29
1½" - ¾"	---	---	5	10	1	2	4	8	---	---	50

Please note that the calculated values in columns "D" and "E" for "Percent Loss" and "Weighted Percent Loss" are expressed to the nearest 0.1%. The total weighted loss for the entire sample is expressed to the nearest 1%.

Sample Report – – Soundness of Fine and Coarse Aggregates

Date: _____ Project: _____

Material: _____ Solution: _____ No. of Cycles: ____

Source: _____ Tested By: _____

Sieve Size	Grading of Original Sample, %	Weight of Test Fractions Before Test, g	Percent Passing Designated Sieve After Test	Weighted Percent Loss
Soundness Test of Fine Aggregate				
Minus No. 100	6	---	---	---
No. 50 to No. 100	11	---	---	---
No. 30 to No. 50	26	100	4.2	1.1
No. 16 to No. 30	25	100	4.8	1.2
No. 8 to No. 16	17	100	8.0	1.4
No. 4 to No. 8	11	100	11.2	1.2
3/8" to No. 4	4 [A]	---	11.2 [A]	0.4
Totals	100	---	---	5
Soundness Test of Coarse Aggregate				
2½" to 2" 2825 g 2" to 1½" 1958 g **2½" to 1½"Fraction:**	20	4783	4.8	1.0
1½" to 1" 1012 g 1" to ¾" 513 g **1½" to ¾" Fraction:**	45	1525	8.1	3.6
¾" to ½" 675 g ½" to 3/8" 333 g **¾" to 3/8" Fraction:**	23	1008	9.5	2.2
3/8" to No. 4 298 g	12	298	11.1	1.3
Totals	100	---	---	8

Qualitative Examination of Coarse Sizes

Particles Exhibiting Distress

Sieve Size	Disinteg.		Splitting		Crumbling		Cracking		Flaking		Total No. of Particles Before Test
	No.	%	No.	%	No.	%	No.	%	No.	%	
2½" to 1½"	---	---	2	7	---	---	2	7	---	---	29
1½" to ¾"	---	---	5	10	1	2	4	8	---	---	50

Note [A] Since less than 5% of this size was present in the sample gradation, no testing of this fraction was performed. The percentage loss of the next size was used to calculate weighted loss.

REVIEW QUESTIONS

1. Describe the sample containers that may be used according to this FOP.

2. According to this FOP, what is the acceptable temperature range for the samples while they are immersed in the soundness solutions?

3. If less than 5% of one of the specified sizes is present in the material to be tested, what should be done? How is the weighted percent loss calculated for this size?

4. How much material is required for testing of the 2½ to 1½ inch size fraction? What should be done if the material being tested has less than 5% in the 2 to 1½ inch size?

5. What should be done if the aggregate being tested has both 10% coarser than the ⅜ inch and finer than the No. 4 sieves?

6. For how long are the samples immersed in the soundness solution during each cycle?

7. Describe determination of the evaporation rate.

8. For the ¾ to ½ inch size fraction, what sieve is used to determine loss?

9. Given that the ⅜ inch to No. 4 size constitutes 13% of the material under test, what is the weighted percent loss for this size fraction if the masses before and after testing are 303 g and 286 g respectively?

PERFORMANCE EXAM CHECKLIST

SOUNDNESS OF AGGREGATE BY USE OF SODIUM SULFATE OR MAGNESIUM SULFATE
FOP FOR AASHTO T 104

Participant Name:_____ Exam Date:_____

Procedure

Sample Preparation

Sample obtained by T 2 and reduced by T 248? _____

Fine Aggregate:

1. Passed through a 3/8 inch sieve? _____
2. Washed on a No. 50 sieve? _____
3. Dried to constant mass at 230 ±9° F? _____
4. Sample rough graded to obtain approximately 110 g of each of the following sizes, if required:
   - 3/8 inch to No. 4? _____
   - No. 4 to No. 8? _____
   - No. 8 to No. 16? _____
   - No. 16 to No. 30? _____
   - No. 30 to No. 50? _____
5. If sample contains less than 5% of any specified size, that size not tested? _____
6. Each size sieved a second time to refusal? _____
7. Aggregates sticking in sieve opening discarded? _____
8. 100 ±0.1 g of each size weighed out and put in separated containers? _____

Coarse Aggregate:

1. Material finer than No. 4 removed? _____
2. Aggregate thoroughly washed? _____
3. Dried to constant mass at 230 ±9° F? _____
4. By sieving to refusal, sample separated into the following sizes with masses as indicated:
   - **2½ to 1½ inch 5000 ±500g, consisting of:** _____
     - 2½ to 2 inch 3000 ±300g _____
     - 2 to 1½ inch 2000 ±200g _____
   - **1½ to ¾ inch 1500 ±50g, consisting of:** _____
     - 1½ to 1 inch 1000 ±50g _____
     - 1 to ¾ inch 500 ±30g _____
   - **¾ to ⅜ inch 1000 ±10g, consisting of:** _____
     - ¾ to ½ inch 670 ±10g _____
     - ½ to ⅜ inch 330 ±5g _____
   - **⅜ inch to No. 4: 300 ±5g** _____
5. If sample contains less than 5% of any specified size, that size not tested? _____

Procedure

1. Salt cake in bottom of solution container broken up and stirred? _____
2. Specific gravity of solution checked? _____
3. Each sample immersed to a depth at least ½ inch above its top? _____
4. Kept immersed for 16 to 18 hours? _____
5. After removal from solution, each sample drained 10 to 20 minutes? _____
6. Dried to constant mass at 230 ±9° F? _____
7. Cooled to room temperature: 68 to 77° F? _____
8. Temperature of aggregate checked by thermometer or other acceptable means before placing in sulfate solution? _____
9. Re-immersed and process continued until required number of cycles is completed? _____
10. After final cooling, sample washed by circulating water at 110 ±10° F through the samples in their containers? _____
11. Hot water introduced near bottom and allowed to pass through samples and overflow? _____
12. Impact or abrasion of samples avoided during washing operation? _____
13. Barium chloride used to check completeness of washing? _____
    Note: If barium chloride reacts with lab water, completeness of washing must be determined by other means.
14. Each fraction dried to constant mass at 230 ±9° F? _____
15. Fine Aggregate: Sieved over same sieves used before test and in the same manner? _____
16. Coarse Aggregate: Hand sieved with minimum effort needed, over:
    - 1¼ inch sieve for 2½ to 1½ inch? _____
    - $^{5}/_{8}$ inch sieve for 1½ to ¾ inch? _____
    - $^{5}/_{16}$ inch sieve for ¾ to $^{3}/_{8}$ inch? _____
    - No. 5 sieve for $^{3}/_{8}$ inch to No. 4? _____
17. Material retained on each sieve weighed? _____
18. Calculations for individual loss and weighted loss performed correctly? _____

Comments: First attempt: (Pass/Fail) ___ Second attempt: (Pass/Fail) ___

Examiner Signature _____WAQTC #:_____